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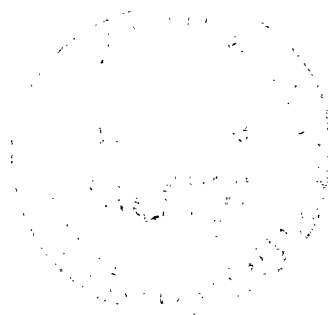


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# Nitriding of Titanium and Titanium– 8-Percent-Aluminum– 1-Percent-Molybdenum– 1-Percent-Vanadium Alloy With an Ion-Beam Source

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**Nitriding of Titanium  
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8-Percent-Aluminum—  
1-Percent-Molybdenum—  
1-Percent-Vanadium Alloy  
With an Ion-Beam Source**

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National Aeronautics  
and Space Administration

Scientific and Technical  
Information Branch



## Summary

Titanium and titanium-8-percent-aluminum-1-percent-molybdenum-1-percent-vanadium alloy (Ti-8Al-1Mo-1V) were nitrided with an ion-beam source of nitrogen or argon and nitrogen at a total pressure of  $2 \times 10^{-4}$  to  $10 \times 10^{-4}$  torr. The treated surface was characterized by surface profilometry, X-ray diffractometry, Auger electron spectroscopy (AES), and microhardness measurements. The tetragonal titanium nitride ( $\text{Ti}_2\text{N}$ ) phase formed in pure titanium and Ti-8Al-1Mo-1V alloy with traces of aluminum nitride (AlN) in the alloy. Two opposite processes competed during the ion-beam-nitriding process: (1) formation of nitrides in the surface layers and (2) sputtering of the nitrided layers by the ion beam. The highest surface hardnesses, of about  $500 \text{ kg mm}^{-2}$  in titanium and  $800 \text{ kg mm}^{-2}$  in Ti-8Al-1Mo-1V, were obtained by ion nitriding with an ion beam of pure nitrogen at  $4.2 \times 10^{-4}$  torr at a beam voltage of 1000 V.

## Introduction

Nitriding is one of the processes used for increasing wear resistance of metals by surface hardening. The method is used especially for steels; it has the advantages that it can be done at relatively low temperatures, it does not require subsequent quenching, and distortion and dimensional changes of the metal part are small. Conventional nitriding of steel is performed in partially dissociated ammonia or by immersing the part in cyanide-cyanate salt baths up to 50 hr (refs. 1 to 4). Besides the long treatment time, the conventional process has the disadvantage of requiring handling of large quantities of ammonia gas or salt bath compounds with related potential environmental hazards. Because of these disadvantages, different nitriding methods were considered. The ion-nitriding process, usually performed by glow discharge, offers the considerable advantages of shorter nitriding times and reduced distortion and growth of the treated parts compared with the conventional process (refs. 5 to 8).

An alternative nitriding method worth investigating is exposing the metal to an ion beam containing nitrogen. The ion-beam technique was used initially by Aisenberg and Chabot (ref. 9) to deposit diamondlike films; Weissmantel reported the formation of iron nitrides in the surface layers ( $0.06 \mu\text{m}$  deep) of iron exposed to a nitrogen ion beam (ref. 10). The ion-beam-nitriding method offers the advantage of treatment in environmentally clean and safe gases (nitrogen or argon). The treatment can be performed in reactors of low background pressure (less than  $10^{-6}$  torr) to reduce the possibility of contamination of the surface by oxygen.

Titanium is a metal suitable for nitriding. It is of interest since formation of hard titanium nitrides ( $1800$  to  $2000 \text{ kg mm}^{-2}$ , ref. 11) in the surface layer of titanium or its alloys can significantly improve their surface hardness and, usually as a result, wear resistance.

The present work investigated the feasibility of nitriding titanium and a titanium alloy with an ion-beam source.

The ion-beam-nitriding experiments (conditions given in table I) were performed to investigate the effect of the following parameters on the nitriding process:

- (1) Beam voltage
- (2) Angle of incidence
- (3) Pressure of nitrogen and argon

## Experimental Procedure

### Ion-Beam Apparatus

The ion nitriding was performed with a 2.5-cm ion-beam source in a 45.72-cm (18-in.) diameter Pyrex bell jar. A schematic drawing of the system is presented in figure 1. The incidence angle (defined here as the angle between the axis of the ion beam and the plane of the target) could be varied between  $0^\circ$  and  $90^\circ$ . In the present work two angles were used,  $30^\circ$  and  $45^\circ$ . The ion-beam voltage could be adjusted up to 1500 V, but only a maximum of 1000 V was used. The ion-beam current was 70 mA during all experiments.

The bell jar was initially pumped down to about  $5 \times 10^{-7}$  torr with a liquid-nitrogen trapped-oil-diffusion pump. Ultra-high-purity argon and nitrogen were then introduced into the bell jar until the desired pressure and gas mixture were obtained. The composition of the gas mixture was determined by the partial pressures of the gases as given in table I. The pressure in the bell jar was measured with an ion gage throughout the experiment. It was not possible to initiate a discharge in the source in pure nitrogen at pressures below  $4 \times 10^{-4}$  torr. For example, it was necessary to add argon at a partial pressure of  $1 \times 10^{-4}$  torr to nitrogen at a partial pressure of  $1.4 \times 10^{-4}$  torr in order to start and sustain the ion beam. All samples were treated for 6 hr and cooled to room temperature in vacuum.

### Materials

The ion-beam nitriding was performed on sheets of 99.94-percent high-purity titanium, 0.163 cm (1/16 in.) thick and of Ti-8Al-1Mo-1V alloy, 0.10 cm (0.04 in.) thick. Prior to nitriding, the samples  $1.9 \times 1.9 \text{ cm}$  (3/4 by 3/4 in.) were polished with  $1 \mu\text{m}$  diamond paste and cleaned in acetone and Freon.

TABLE I. - ION-BEAM-NITRIDING CONDITIONS

Sample	Pressure		Beam voltage, V	Incidence angle, deg	Maximum temperature, °C	Sputtering rate, $\mu\text{m hr}^{-1}$
	Argon	Nitrogen				
Titanium						
1	$1.5 \times 10^{-4}$	$0.5 \times 10^{-4}$	1000	45	390	3.3
2	.5	9.5	500	45	190	.2
4	.5	9.5	1000	45	392	1.8
5	0	10.0	↓	30	340	.8
6	1.0	1.4		↓	350	1.4
7	0	4.2			350	.9
Ti-8Al-1Mo-1V						
5	0	10.0	↓	↓	340	1.0
6	1.0	1.9			350	1.6
7	0	4.2			350	.8

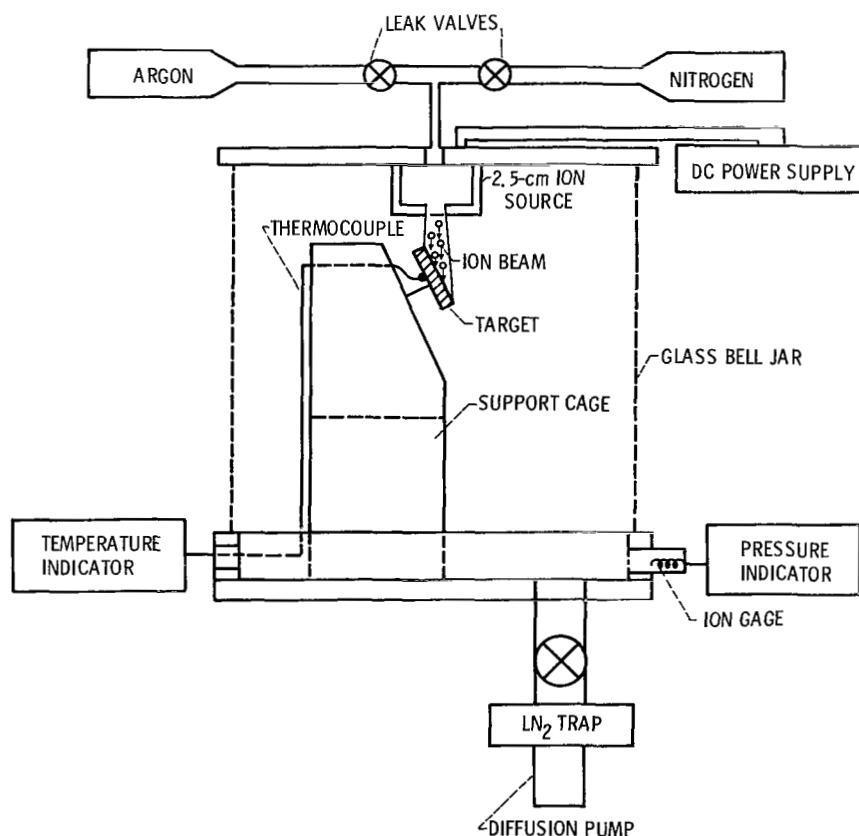


Figure 1. - Ion-beam apparatus.

### Temperatures

During the ion-beam nitriding the samples were fixed on the holder by spring clips. The holder was a stainless steel plate 7.62 cm (3 in.) in diameter and 3 mm thick. A thermocouple was welded at the center of the holder on its back. The whole surface of holder and samples was exposed to the ion beam. The measurements indicated that the equilibrium temperature was reached within 50

to 60 min from the start of the ion beam, and afterwards the temperature remained constant during nitriding. Since most of the temperature losses were by radiation, it could be assumed that, with this experimental setup, the measured temperature was that of the bulk of the sample holder and close to the bulk temperature of the samples. Table I indicates the maximum temperature measured in each experiment.

## Characterization

As a result of the exposure to the ion beam the samples were etched by sputtering. The profile of the etched surface and the depth of sputtering were measured with a profilometer. The depth of sputtering was determined by measuring the height of the step formed by the holding clip, which was masking a part of the sample.

X-ray diffractometry was used to identify the phases formed during the nitriding process. The diffractograms of the nitrided samples were compared with the diffractograms of the original metals. Since the intensity of the new diffraction lines was generally very weak, each X-ray scan was repeated several times to differentiate between peaks and background noise.

Depth profiling by sputtering together with Auger electron spectroscopy (AES) gave the composition at different depths of the nitrided surfaces. The AES measurements were made in a baked ultra-high-vacuum system which was ion pumped to  $<2 \times 10^{-10}$  torr and then backfilled with ultra-high-purity argon to  $3.8 \times 10^{-5}$  torr. The sputtering was performed with 3-kV  $\text{Ar}^+$  ions at an emission current of 10 mA. A commercial single-pass cylindrical mirror analyzer with an integral electron gun was used. The gun was operated at 2 kV and 2 mA. The detection system was operated in the  $\text{dN/dE}$  mode with a modulator peak-to-peak voltage of 1 V. The diameter of the spot was  $37 \mu\text{m}$ .

The microhardness of the ion-beam-nitrided surface was measured with a diamond Knoop indenter at loads of 100 to 200 g.

## Results and Discussion

After ion-beam nitriding, the original mirror-smooth surface has a dull appearance similar to that of metal exposed to sandblasting. A typical profilometer trace of a sample ion nitrided for 6 hr is shown in figure 2. The roughness of the nitrided surface as compared with the original surface can clearly be seen. The surfaces exposed to the ion beam were sputtered at a high rate of up to  $3.3 \mu\text{m hr}^{-1}$ . The sputtering rates for different ion-beam conditions are given in table I. The results indicate that the sputtering rate increases strongly when the beam voltage increases from 500 to 1000 V and is directly related to the partial pressure of argon, as expected from previously reported results (see figs. 6-3 and 6-5 of ref. 12). This indicates that the sputtering process is competing with the nitriding process by sputtering away the nitrided layers at a significant rate.

### Phase Identification

Figure 3 presents the X-ray diffractogram of the original titanium metal and a diffractogram typical of samples 4 to 7 of ion-beam-nitrided titanium. The

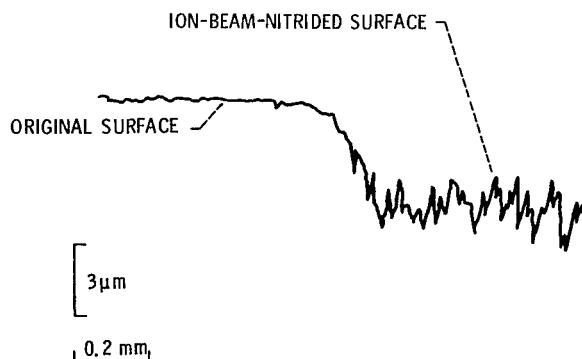


Figure 2 - Profilometer trace of ion-beam-nitrided titanium sample. Argon pressure, 0; nitrogen pressure,  $4.2 \times 10^{-4}$  torr; treatment time, 6 hours.

diffractograms are shown without the background noise. The diffractogram of sample 1 was identical to that of the original metal, while the diffractogram of sample 2 showed a widening of the original X-ray peaks without the occurrence of any new peak. This indicates that none of these samples contained nitride phases. In the case of sample 1, it is possible that some nitride phases formed in the external layers of the exposed metal, but, because of the high sputtering rate, the sputtering was faster than the nitride formation and no nitrided layer was left after the ion-beam-nitriding process. In the case of sample 2, it is possible that either the ion-beam voltage of 500 V or the bulk temperature of the sample,  $190^\circ \text{C}$  was too low for nitride formation. It is possible that, if the sample were heated independently to a higher temperature, nitride formation would occur even at an ion-beam voltage of 500 V.

As shown by the lower curve in figure 3, several weak peaks occurred in the samples nitrided at an ion-beam voltage of 1000 V and nitrogen pressures above  $1.4 \times 10^{-4}$  torr. All peaks in this curve are identified in table II. As a result of the ion-beam nitriding, the tetragonal ( $\text{Ti}_2\text{N}$ ) 6T phase (ref. 13) formed in the titanium matrix. However, one X-ray line (at  $2\theta = 36.8^\circ$ ) could not be attributed to the tetragonal ( $\text{Ti}_2\text{N}$ ) 6T phase but to either the cubic ( $\text{TiN}$ ) 8E (ref. 14) or tetragonal ( $\text{Ti}_2\text{N}$ ) 12U phase (ref. 15). This indicates that a small amount of either phase was also formed during the ion-beam-nitriding process. Similar behavior was observed in the X-ray diffractograms of the nitrided titanium alloy. In addition, two weak lines corresponding to the strongest lines of  $\text{AlN}$  could be identified, indicating the formation of this phase in the nitrided alloy.

### Composition of Ion-Nitrided Layer

Figure 4 presents the AES spectra of nitrogen and titanium according to McGuire (ref. 16). The overlap of the peaks at 381 eV (N) and 381 to 386 eV (Ti) makes

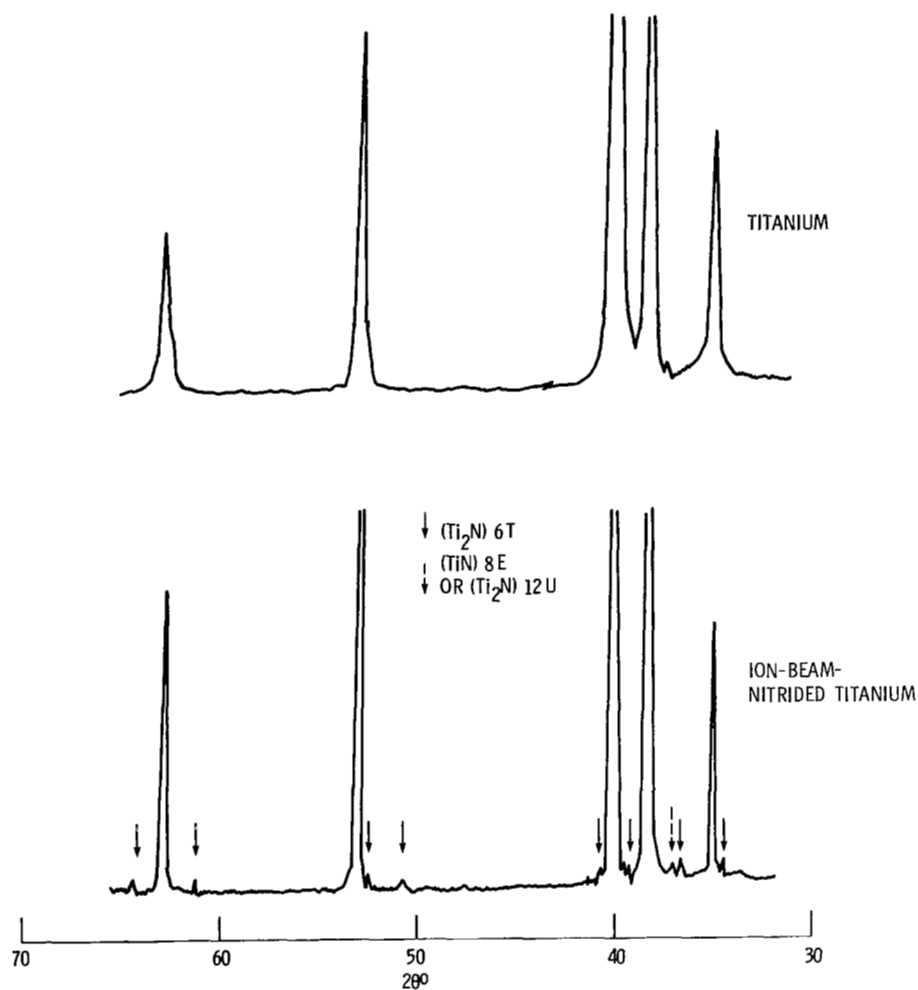


Figure 3. - X-ray diffractograms. (Peaks are identified in table II.)

TABLE II. - X-RAY DIFFRACTOMETER LINES OF ION-BEAM-NITRIDED TITANIUM

[Sample 7.]

2θ, deg	d <sub>exp</sub> , Å	d <sub>theor</sub> , Å	hkl (Ti)	hkl (Ti <sub>2</sub> N)6T	hkl (TiN)8E/(Ti <sub>2</sub> N)12U
34.7	2.58	2.586	---	101	-----
35.1	2.55	2.557	010	---	-----
36.3	2.48	2.473	---	200	-----
36.8	2.44	2.44/2.435	---	---	111/112
38.4	2.34	2.342	002	---	-----
39.3	2.29	2.292	---	111	-----
40.2	2.24	2.244	011	---	-----
40.8	2.21	2.211	---	210	-----
51.0	1.79	1.788	---	211	-----
52.3	1.75	1.749	---	220	-----
53.0	1.73	1.726	012	---	-----
61.4	1.51	1.517	---	002	-----
63.1	1.47	1.475	110	---	-----
64.3	1.45	1.448	---	301	-----

direct identification of nitrogen in the nitrided layer difficult. However, the occurrence of nitrogen in the titanium matrix should change both the shape and the relative peak-to-peak heights of the titanium peaks. Thus, the shapes of the peaks between 360 and 420 eV and their change during depth profiling will give a qualitative indication of the existence of nitrogen in the nitrided layers. Since the shape of the AES peaks may be affected by the measuring apparatus and conditions in AES, a spectrum is shown in figure 5 (bottom curve). Though the relative peak-to-peak heights are different from those in figure 4, their shapes are essentially the same.

The top curve in figure 5 presents the AES spectra of the ion-beam-nitrided surface of sample 4 after sputter cleaning for 3 min in the Auger apparatus. The 381-eV peak is shifted to 383 eV and is less resolved than in untreated titanium, and the peak-to-peak height of the 383- to 386-eV peak is much higher than the peak-to-peak height of the 417-eV peak. This is in contrast to the spectrum of the untreated titanium shown by the bottom

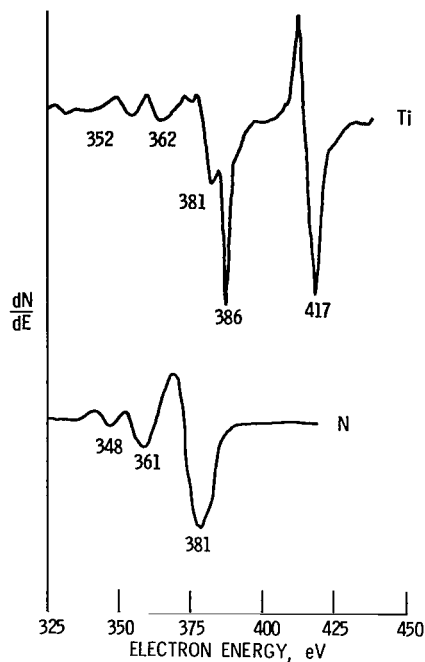


Figure 4. - AES spectra of titanium and nitrogen.

curve in figure 5, where the peak-to-peak height of the 381- to 386-eV peak is smaller than the peak-to-peak height of the 417-eV peak. The shape of the 383- to 386-eV peak is also similar to that of the 381-eV peak of nitrogen (lower curve in fig. 4). This indicates the existence of nitrogen together with titanium in the measured nitrated layer.

A similar AES spectrum was reported for thermally nitrated titanium in reference 17. The center curve in figure 5 presents the AES spectrum of the same sample after sputtering for 30 min at 10 mA and 2 kV. The shape of the spectrum changes toward that of untreated titanium but still indicates the occurrence of nitrogen at the corresponding depth. Figure 6 presents the AES spectra of the same sample 4 at different depths. The top and middle curves in figure 6 are the same as the top and middle curves in figure 5; however, the bottom curve in figure 6 is the spectrum after 45 min of sputtering. Figure 7 presents the AES spectra of sample 5 after initial sputter cleaning (top curve), after 30 min of sputtering (middle curve), and after 40 min of sputtering (bottom curve). The sputtering conditions were the same as those for sample 4 (fig. 6). The ratio of the peak-to-peak heights of the 386-eV peak to that of a 417-eV peak in the bottom curve in figure 6 is higher than the corresponding

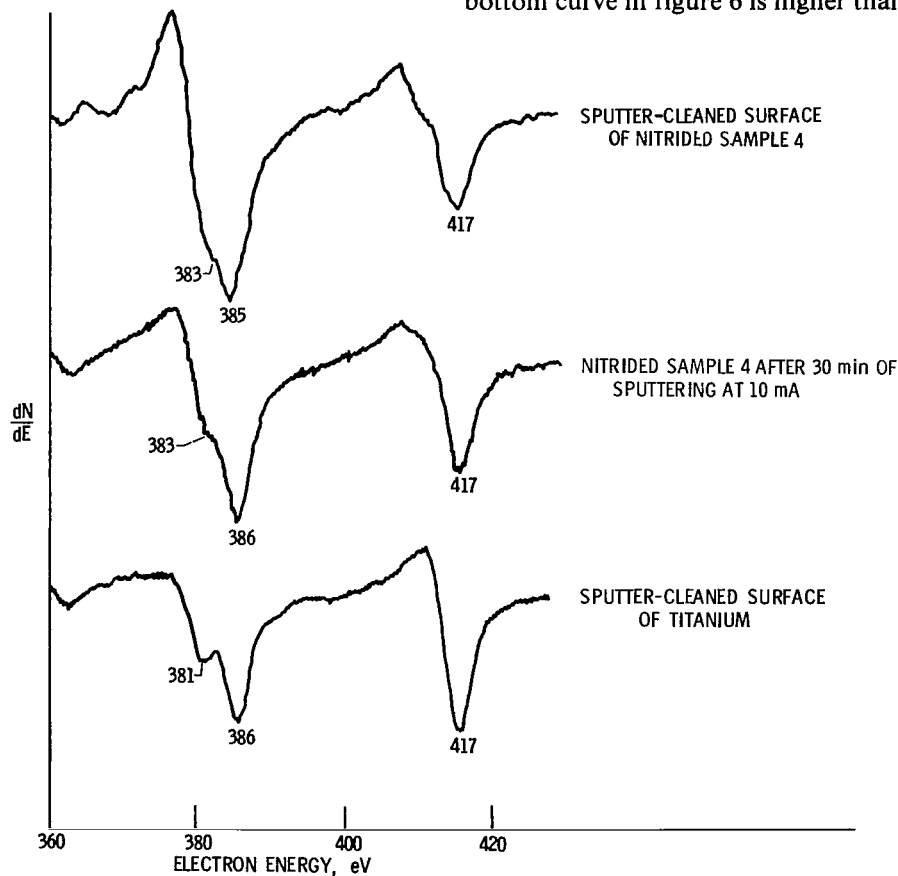


Figure 5. - AES spectra of ion-beam-nitrated titanium (sample 4) and of pure titanium.

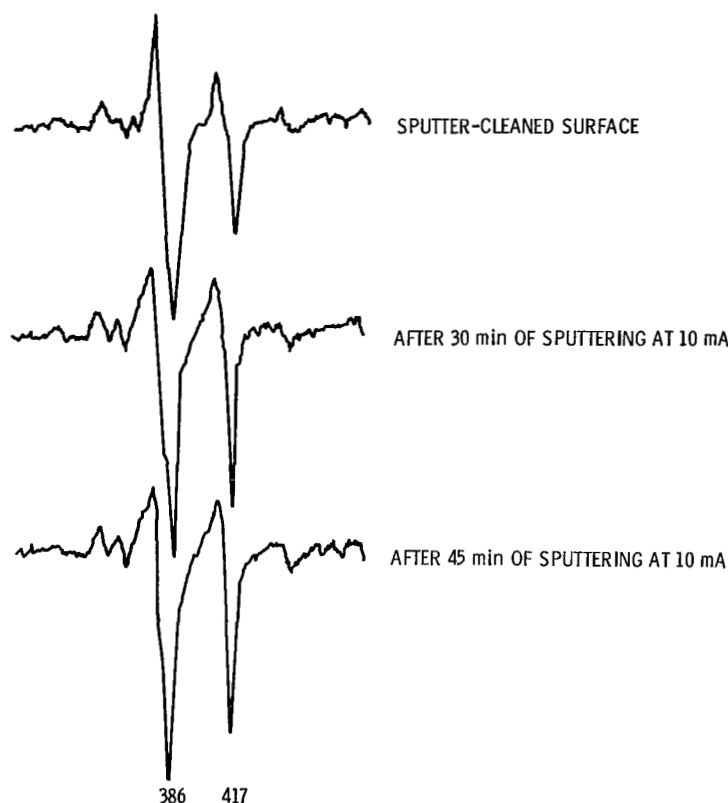


Figure 6. - AES spectra of ion-beam-nitrided titanium (sample 4).

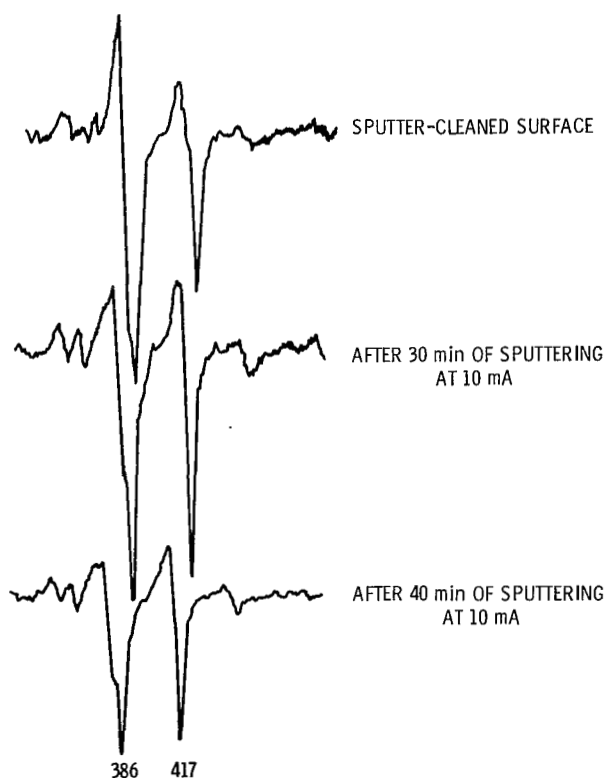


Figure 7. - AES spectra of ion-beam-nitrided titanium (sample 5).

ratio for the untreated titanium (fig. 5, bottom curve). This indicates that, in sample 4 the surface exposed after 45 min of sputtering contained significant amounts of nitrogen. In contrast, the ratio of the peak-to-peak heights of the 386-eV peak to that of 417-eV peak in the bottom curve in figure 7 is essentially the same as that for the untreated titanium, indicating that the titanium of sample 5 did not contain nitrogen at the depth reached after 40 min of sputtering. (The spectrum at the bottom in fig. 7 is the same as that of untreated titanium (fig. 5, bottom).) Since the depth profiling was performed under the same conditions for samples 4 and 5 of titanium, the AES results indicate that sample 4 was nitrided to a lower depth than sample 5.

### Microhardness

Since the titanium nitride phases are much harder (Knoop microhardness KHN at 100 g  $\approx 1800 \text{ kg mm}^{-2}$ , ref. 11) than the metal phases, it is expected that the surface hardness will be increased by nitriding. The microhardness of the surface layers was measured by applying a Knoop indenter normal to the surface. The microhardnesses at loads of 100 and 200 g are given in table III. The microhardnesses of the untreated titanium and titanium alloy are also given for comparison. Table III indicates that the surface microhardness at 100 g increases by ion nitriding from 230 to about 500 kg



TABLE III. - KNOOP  
MICROHARDNESS OF ION-  
BEAM-NITRIDED TITANIUM  
AND Ti-8Al-1Mo-1V

Sample	Knoop microhardness, KHN, kg mm <sup>-2</sup>	
	At 100-g load	At 200-g load
Titanium	230 ± 10	220 ± 10
1	230 ± 10	220 ± 10
2	230 ± 10	220 ± 10
4	506 ± 60	368 ± 40
5	353 ± 20	278 ± 10
6	293 ± 10	270 ± 10
7	497 ± 40	368 ± 20
Ti-8Al-1Mo-1V	370 ± 20	365 ± 20
5	671 ± 50	680 ± 40
6	474 ± 20	470 ± 20
7	798 ± 50	672 ± 40

mm<sup>-2</sup> for pure titanium and from 370 to about 800 kg mm<sup>-2</sup> for Ti-8Al-1Mo-1V alloy. No increase in surface hardness was observed for the sample treated with an ion-beam of 500 V (sample 2) or for the sample etched at the high rate of 3.3  $\mu\text{m hr}^{-1}$  (sample 1). This indicates that no measurable hardening was produced by the ion bombardment of the surface. Tables I and III indicate that, for the samples treated with an ion beam incident at an angle of 30°, the surface hardness increased with increasing pressure of nitrogen from  $1.4 \times 10^{-4}$  to  $4.2 \times 10^{-4}$  torr. A further increase in the nitrogen pressure to  $10 \times 10^{-4}$  torr decreased the resulting surface hardness. It is possible that, with the increase of the pressure, recombination of the nitrogen particles (ions and atoms) occurred in the ion beam, and thus the concentration of the nitriding particles decreased. The nitride formation in the metals was reduced, and, as a result, the obtained surface hardness was smaller (sample 5 compared with 7).

Both AES and microhardness results indicate that the nitriding of sample 4 was more effective than that of sample 5. Both samples were nitrided at the same ion-beam voltage and total pressure. However, the temperature of sample 4 was about 50° C higher than that of sample 5 as a result of a larger incidence angle. Since the depth of the nitrided layer is essentially controlled by a diffusion process, the temperature of the sample is very important. In the ion beam treatment two opposite processes are competing:

(1) Nitrogen atoms are introduced in the surface layers of the metal and diffuse inside to form metal nitrides.

(2) The surface layers, having the highest concentration of nitrogen, are removed by sputtering of the ion beam.

The first process has to be faster than the second one to obtain a nitrided surface after ion-beam treatment. The competition of the two processes can explain the higher surface hardness obtained for sample 4 than for sample 5 and the lack of nitriding observed in sample 2, whose bulk temperature reached only 190° C. Though the sputtering rate was higher for sample 4 than for sample 5, the rate of diffusion of nitrogen and the formation of nitride was much faster in sample 4 because of the higher bulk temperature. The relatively low bulk temperature of sample 2 prevented any formation of nitrides in the surface layers of the metal. By increasing the temperature of the substrate above 350° C independently of the power of the ion beam, one may achieve nitriding at beam voltages below 1000 V and reduced sputtering rates.

## Conclusions

After titanium and Ti-8Al-1Mo-1V were treated with an ion beam of nitrogen or nitrogen and argon, it was found that

1. It is possible to ion nitride the metals by using ion beams with a voltage of 1000 V.
2. Sputtering of the surface layers by the ion beam at a rate of 0.8 to 1.8  $\mu\text{m hr}^{-1}$  competes with the nitriding process.
3. Tetragonal Ti<sub>2</sub>N phase is formed in the ion-beam-nitrided titanium and its alloy with traces of hexagonal AlN in the alloy.
4. As a result of the nitriding the surface microhardnesses of both the pure titanium and the alloy are doubled.
5. The highest microhardness is obtained with an ion beam of pure nitrogen at a pressure of about  $4 \times 10^{-4}$  torr.

Lewis Research Center  
National Aeronautics and Space Administration  
Cleveland, Ohio, December 6, 1982

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